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EFFECT OF DOUBLE THERMAL PRE-AGEING ISOTHERMAL TREATMENT ON VICKER'S MICRO-HARDNESS OF Ti6Al4V ALLOYS FOR DENTAL IMPLANT APPLICATIONS

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Abstract

Ti alloys have found wide range of engineering applications in different sectors due to excellent properties they possess. Ti6Al4V is a class of Titanium alloys that has been referred to as a bio-alloy due to its biocompatibility, good osteointegration with no adverse tissue reaction. Microhardness measurements have been used in non-destructive testing for qualitative analysis and as a predictive tool in most processing conditions involving Ti6Al4V alloys. Vickers' microhardness (Hv) test was conducted on double thermal treated samples and untreated samples to investigate the effect of pre-ageing temperature and ageing time on the hardness of Ti6Al4V alloy. A quick screening method for selecting some of the most effective factors for further experimental design based on design of experiment (DOE) was presented. The model uses two level factorial design of 16 runs to evaluate the effect of four factors namely - A: Pre-ageing time, B: Pre-ageing temperature, C: Ageing time and D: Ageing temperature on Vickers' microhardness response. The two-level factorial design was selected for the model because it could model interaction effects. The Hv of the untreated specimen was 372.67, however, there was significant improvement in the microhardness values of double thermal treatment samples. The microhardness at pre-ageing temperature of 300^{o}C and soaking time of 2 Hours was 779, while at 8 Hours the value was 831.67Hv; this corresponds to an increase in Hv of 6.76%. The interaction of BC, BD and CD were found to be significant, while optimization of the process parameters was achieved at a 'Desirability' of 0.971.

Key words: *Double thermal ageing, Pre-ageing, Microhardness, Two-level factorial, Ti6Al4V, Bio-alloy.*

1.0 Introduction

Titanium and its alloys have played vital roles in many engineering applications in recent times, owing to a number of reasons which include high tensile strength and modulus, good corrosion resistance, high temperature capabilities, good creep resistance, light weight and above all excellent biocompatibility [1]. These attributes as we have mentioned had endeared Ti alloys to engineers and researchers to continuously find a way to improve on their properties and extend their functionalities [2]. Among the numerous alloys of Ti, Ti6Al4V alloys stands out because of its good corrosion resistance, light weight, excellent mechanical properties and biocompatibility [3].

Ti6Al4V alloy is of particular interest because of the fact that its properties could be modified or enhanced through heat treatment processes. Two major phases which are the Alpha and Beta phases play major roles in improving the strength and hardness of the alloy. Aluminium plays the role of Alpha stabilizer while Vanadium plays the role of Beta stabilizer; and on quenching the alloy the stabilizers help in retaining the desired phases which helps in improving the strength [4]. The presence of different phases which varies as the temperature varies is one of the mechanisms of improving the hardness of Ti alloys. Hardness is an important mechanical property when Ti alloys are to be used in some biomaterial applications [5].

Hardness is one of the most important and quick method of evaluating mechanical properties of metals and alloys. Hardness offers a qualitative assessment of the tensile and yield strength of alloys such as Ti alloys. Microhardness measurements have been used in non-destructive testing for qualitative and as a predictive tool in most processing conditions involving the alloys [6]. Keist and Palmer[7], investigated the use of microhardness measurement as a predictive tool for additive manufactured Ti6Al4V alloy components to check the effects of processing parameters and post processing heat treatment on the mechanical properties of the alloys.

Heat treatment operations have been reported to improve the hardness of different types of Ti alloys. Poondla *et al.* [4], investigated the influence of alloying composition and secondary processing on the hardness properties of pure Ti and Ti6Al4V alloys and concluded that there was a remarkable increase in the hardness value of Ti6Al4V due to the alloying element and secondary processing. Masete*et al.*, [8], investigated the effect of ageing temperature, ageing time and cooling medium on the hardness of Ti6Al4V alloy, they discovered that furnace cooling after ageing for 30 minutes resulted in better homogenization than solution treated and water quenched Ti6Al4V alloy. Furthermore, they observed that higher ageing temperatures above 700^{o}C was detrimental to the microhardness.

Several processing techniques are available for the production of Ti6Al4V alloys such as laser melting techniques, direct metal laser sintering, powder metallurgy, explosive forming and other advanced processing routes. Heat treatment processes such as solution treatment and age-hardening techniques have been conducted to improve the mechanical properties such as hardness based on such processing methods with improvement in properties reported [9; 10]. Hardness evaluation of Ti6Al4V alloys have been shown to have a wide range of variability due to orientation of phases [7]. Admittedly, the processing techniques for manufacturing of Ti6Al4V alloys and the heat treatment processes have a wide array of parameters and factors which can influence the mechanical properties such as the microhardness [11]. Use of statistical tools such as design of experiment have been used to optimize responses of Ti based alloys. This enables a more courteous and frugal approach to experimentation, while improving on the measured responses [6; 12].

Ti alloys have been applied in different industries and field of applications due to their excellent properties. Ti6Al4V alloys have been deployed in aerospace industry, petrochemicals, oil and gas, marine industries and in the medical applications as implantable materials [13]. In fact, some scholars have referred to Ti6Al4V alloys as bio-alloys to underscore the importance of the alloy as biomaterial [14]. The alloy has been used as dental implant, prosthetic hip replacement due to good osteointegration, no adverse tissue reaction near implants and good biocompatibility [15]. The objectives of this research work are to investigate the influence of double thermal ageing heat treatment on the hardness of Ti6Al4V alloys for biomaterial applications; and use of two-level factorial design for screen of effects of various factors on microhardness response optimization.

2.0 Materials and Method

2.1 Materials

Titanium alloy (Ti6Al4V) used for this research was obtained from Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology, South Africa.

2.2 Equipment

The equipment used in this research include: Heat treatment furnace, Vickers micro-hardness testing machine, precision cutter, polishing machine.

2.3 Material Preparation

The as received Ti6Al4V alloy (ASTM grade 5) used for this research has dimension of 70 mm ×70 mm×3 mm. The chemical compositions of the as-received Ti6Al4V alloy are shown in Table 1. The Ti6Al4V alloy plate was cut using silicon carbide blade and machined into hardness and wear standard test samples under flowing cooling condition. All the samples were manually polished using silicon carbide emery paper down to 1200 grade to achieve a fine surface finish. In preparation for the heat treatment operations, the samples were thoroughly degreased, cleaned and properly dried.

Table 1: The chemical composition of as-received Ti6Al4V alloy

Ti	Al	V	C	Fe	O	N	H
89.62 %	6.1 %	4 %	0.004%	0.160 %	0.106 %	0.008 %	0.002%

2.4 Heat Treatment of Titanium alloy

Double thermal ageing heat treatment cycle was adopted for the treatment of Ti alloys used for experiment. 32 specimens were used for the heat treatment process, while two specimens were used for the control. The double thermal aging process follows the outlined cycle as stated: the first stage of the double thermal aging is the solution heat treatment (SHT) at 960^{o}C for 1 hour, quenched in warm water at 65^{o}C. The quenched samples were pre-aged at 150^{o}C for 2 hours in the oven; the procedures for the first stage are summarized as (SHT, quenching and pre-aging). The third stage-the thermal treatment, involves charging the specimen in another oven at temperature of 480^{o}C while varying the soaking times for (2, 4, 6, 8 hours). The heat treatment cycles are repeated for temperatures of 200°C, 250°C, and 300°C.

2.5 Hardness Test

The heat-treated samples and the control (non-heat treated) samples were subjected to hardness test using Vickers micro hardness tester. Three replicate measurements were taken for each specimen and the average Hardness Value [8] was recorded.

2.6 Two Level Factorial Design

Design of Experiment (DOE) was used in modelling the responses of four different factors based on two level factorial design. The two-level factorial design was adopted as a quick screening for multi-factor effect estimation. Optimization of the response based on the two-level factorial was equally performed based on the target goal. Table 2 is the design matrix table showing the factors and levels for the two-level factorial design.

Table 2: Two Level Factorial Design Matrix

Standard	Run	A: Pre-ageing time	B: Pre-ageing temp	C: Ageing time	D: Ageing temp
16	1	2	300	8	480
11	2	1	300	2	480
5	3	1	150	8	400
7	4	1	300	8	400
8	5	2	300	8	400
14	6	2	150	8	480
3	7	1	300	2	400
13	8	1	150	8	480
2	9	2	150	2	400
10	10	2	150	2	480
15	11	1	300	8	480
6	12	2	150	8	400
9	13	1	150	2	480
4	14	2	300	2	400
12	15	2	300	2	480
1	16	1	150	2	400

3.0 Results

Figures 1 shows the variation in Vickers micro-hardness of double thermally treated Ti6Al4V alloys with different pre-aging treatment temperatures of 150, 200, 250 and 300^{o}C and varying aging time of 2, 4, 6, 8 Hours.

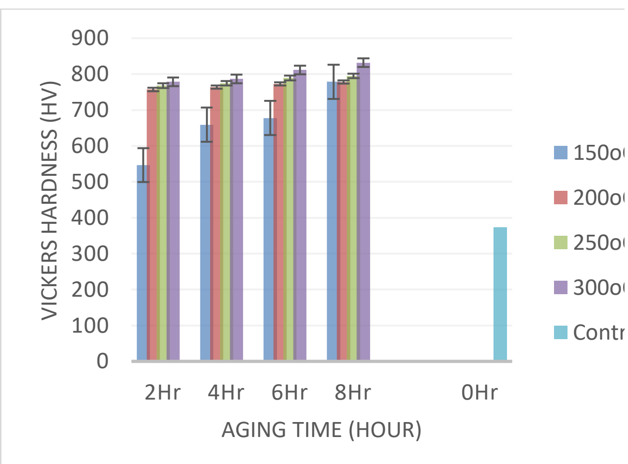

Figure 1: Variation of Vickers Hardness of Ti6Al4V alloys as A Function of Pre-aging Temperature and Aging Time.

From the graph it was observed that the pre-aging treatment temperature, significantly improved the Vickers micro hardness Value of the double thermal heat treated Ti6Al4V alloy[9]. At pre-aging temperature of 150^{o}C, the Hv value was 546.67 Hv and at 300^{o}C the Hv was 779 Hv; this corresponds to increase of 42.50% in Hardness. Similarly, it could further be observed that, there was concomitant increase in Hardness as a result of variation in soaking time of the pre-aged Ti6Al4V alloy. The observed hardness at pre-ageing temperature of 300^{o}C and soaking time of 2 Hours was 779Hv while at 8 Hours the value was 831.67Hv; this corresponds to an increase in Hv of 6.76%. From the plot we could deduce that Vickers hardness increases with increase in pre-aging temperature and aging time. The influence of the double thermal aging heat treatment method on the Vickers hardness of Ti6Al4V alloy was greatly accentuated when we compare the Hv of the untreated Ti6Al4V alloy to those of the treated specimen. The Hv of the untreated specimen was 372.67, while the Hv of Ti6Al4V alloy solution heat treated at 960^{o}C for 1 hour; quenched in warm water at 65^{o}C, pre-aged at 300^{0}C for 2 hours and aged at 480^{o}C for 8 hours was 831.67Hv; this corresponds to an increase in 123.17% in Hardness. This is in agreement to [3] that stated that improvement in properties were as a result of microstructural modification of the Ti alloy due to heat treatment. The ageing treatment performed on the Ti6Al4V alloy owes it improvement in hardness to precipitation of second phase particles that finely distributed in the predominant phase. Sarma *et al.* [3] opined that the transformation of α martensite into finely dispersed precipitate of β phase results in incremental hardness of Ti alloys.

From the Design of Experiment (DOE) two level factorial design, the Analysis of Variance (ANOVA) table showing the observed statistics is presented in Table 3. From the design, it was observed that the model was significant and could be used to predict the experimental behaviour for the Ti6Al4V alloy. The R – Squared value of 0.9858 indicates that the response was linearly corelated to the factors. The predicted R-Squared of 0.7734 is in reasonable agreement with Adj R-Squared of 0.9468 as obtained from the ANOVA statistics. The model regression equation is shown in Table 4. The final equation in terms of actual factors can be used in predicting the Vickers' microhardness of the Ti6Al4V alloy. The two-level factorial design is a quick tool for predicting the influence of individual factors and factor interactions on the measured response [11].

Table 3: Analysis of Variance (ANOVA) for Selected Factorial Model

Source	Sum of Squares	df	Mean Square	F - Value	Prob > F	
Model	84695.34	11	7699.58	25.31	0.0034	significant
A-Pre-ageing time	7441.22	1	7441.22	24.46	0.0078	
B-Pre-ageing temp	28873.66	1	28873.66	94.90	0.0006	
C-Ageing time	23330.27	1	23330.27	76.68	0.0009	
D-Ageing temp	8915.61	1	8915.61	29.30	0.0056	
AB	403.51	1	403.51	1.33	0.3136	
AC	297.13	1	297.13	0.98	0.3790	
AD	14.01	1	14.01	0.046	0.8406	
BC	4611.43	1	4611.43	15.16	0.0176	
BD	4796.60	1	4796.60	15.77	0.0165	
CD	2960.18	1	2960.18	9.73	0.0356	
BCD	3051.73	1	3051.73	10.03	0.0340	
Residual	1216.99	4	304.25			
Cor Total	85912.33	15				

Table 4: Final Model Equation in Terms of Actual Factors

Microhardness	=
Constant terms	Actual Factors
-1473.21167	
+67.04333	* Pre-ageing time
+7.05992	* Pre-ageing temp
+235.80521	* Ageing time
+4.11162	* Ageing temp
-0.13392	* Pre-ageing time * Pre-ageing temp
-2.87292	* Pre-ageing time * Ageing time
+0.046781	* Pre-ageing time * Ageing temp
-0.75064	* Pre-ageing temp * Ageing time
-0.013444	* Pre-ageing temp * Ageing temp
-0.45861	* Ageing time * Ageing temp
+1.53451E-003	* Pre-ageing temp * Ageing time * Ageing temp

From the two level factorial design, different parameters could be estimated. Parameters such as 'All Factors Effect', 'Interaction', contour plot' and many more could be estimated. All the factors had positive effect on the Vickers' microhardness property of the double thermal age-hardened Ti6Al4V alloy for dental implant applications. The factors as coded to actual factors were A: Pre-ageing time, B: Pre-ageing temperature, C: Ageing time, D: Ageing temperature. From the two level factorial of Design of Experiment (DOE), it was observed that all the four factors positively affects the Microhardness value of the Ti6Al4V double thermal age-hardened alloy. Figure 2 shows all factors plot for the two level factorial design. Factors B and C had more significant influence on the Vickers' microhardness value.

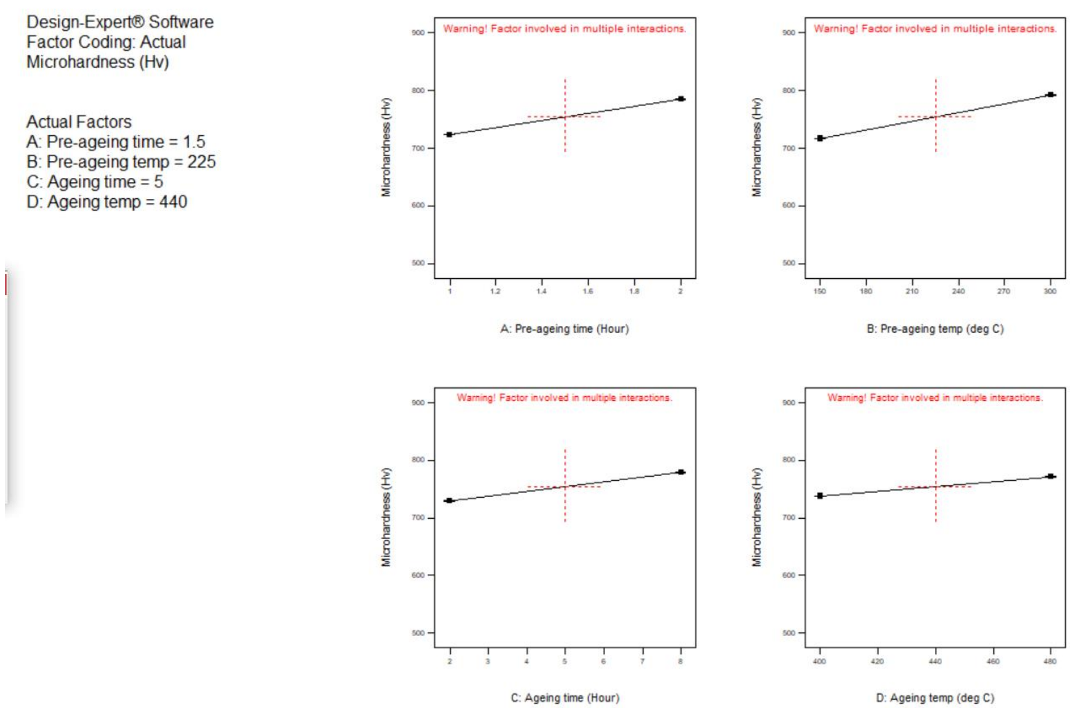

Figure 2: All Factor Plot for Two Level Factorial Design

The interaction effects of B: Pre-ageing temperature and C: Ageing time (BC), B: Pre-ageing temperature and D: Ageing temperature, C: Ageing time and D: Ageing temperature (CD) and BCD all had significant contributions on microhardness; these can be adduced from the ANOVA Table 3. Figure 3show the interactions of factors BC and BD.

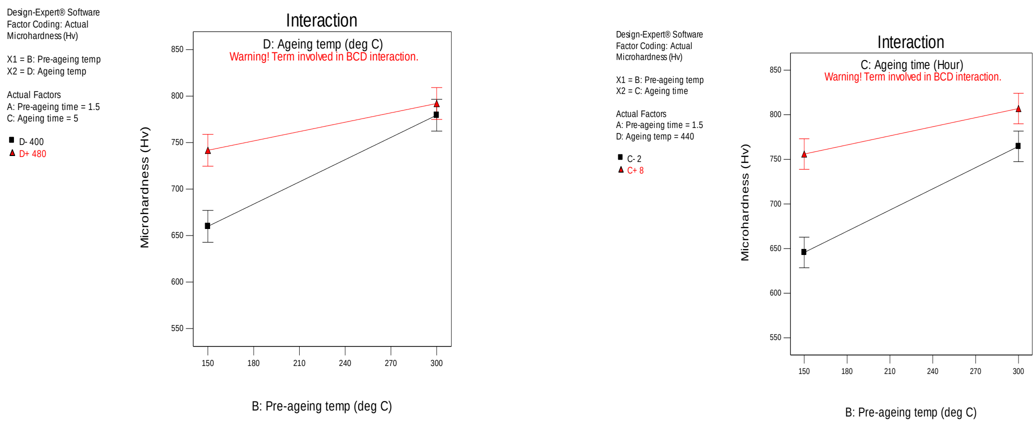

Figure 3: Interaction Plots a) Factor B and C; b) Factor B and D

The contour plot of the factors interactions for BC and BD are shown in the Figure 4. The contour depicts the interaction of different factors with each other on a 2-D surface with contours showing the responses with change in factor level.

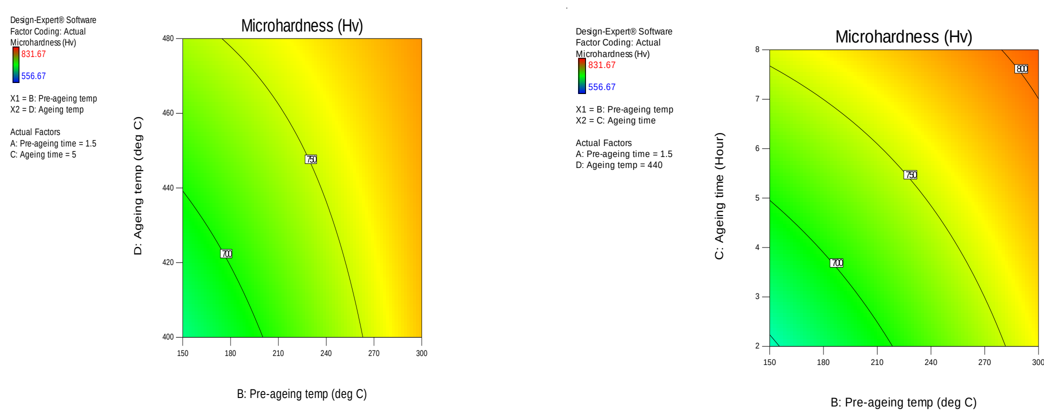

Figure 4: Contour Plots (a) Factor B and C; (b) Factor B and D

The 3D Surface plot of the factor interactions show the 3D surface of the interactions with respect to the measured response - the microhardness. Figure 5 show the 3D Surface plot for BC and BD interaction.

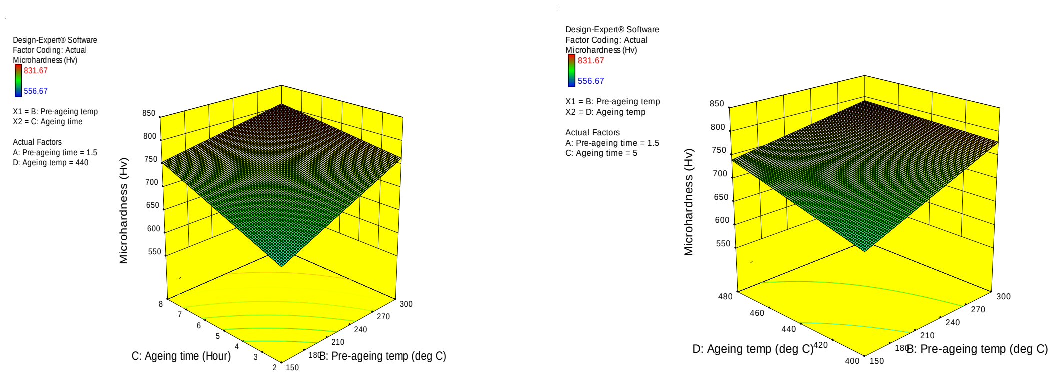

Figure 5: 3 D Surface Plots a) Factor B and C; b) Factor B and D

Optimization of all the factors that affects the Vickers' microhardness were conducted. Optimization goals were in order to obtain the best sets of solution that could best fit the design. From the optimization parameters several solutions were obtained, however solution one was preferred with a Desirability of 0.971. Table 5 shows the parameters in terms of factors, the optimization goals and the obtained optimization values. From the optimization model, it was observed that the ageing time could be reduced to 7 hours to achieve the optimum microhardness. Similarly, the ageing temperature could be reduced to 470°C without it affecting the microhardness response.

Table 5: Optimization Parameters, Goal and Values

Parameter	Goal	Value
A: Pre-ageing time	Target	2 hours
B: Pre-ageing temperature	Target	300 °C
C: Ageing time	Target	7 hours
D: Ageing temperature	Target	470°C
Microhardness	Maximize	819.125 Hv
Standard Error	Minimize	11.9585

The optimized solution for BC interaction graph showing the contour plot of the microhardness, desirability and the Standard Error of the Microhardness is shown in Figure 6. The optimization combinations are expected to lead to reduction in ageing time and ageing temperature; this will lead to improve efficiency of the process and reduction in the cost of energy for the heat treatment operation.

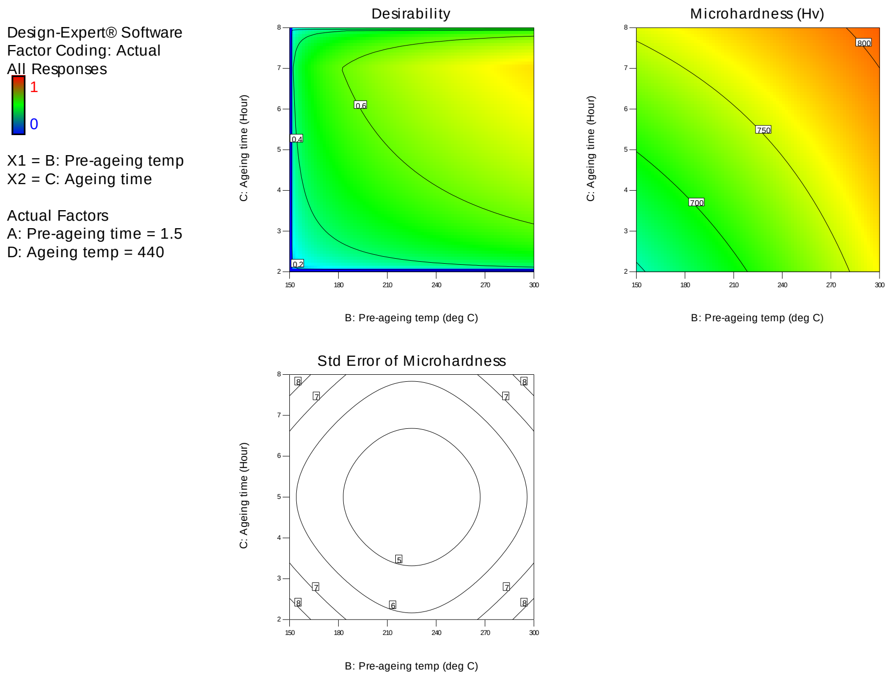

Figure 6: Optimized Microhardness for BC interaction

Conclusion

Effect of double thermal ageing heat treatment process on microhardness of Ti6Al4V alloy for dental implant were investigated. The results showed that the heat treatment process has significant influence on the micro-hardness Hv of the double thermal treated alloys compared to the untreated alloys. The following conclusions were drawn:

i. at pre-aging temperature of 150^{o}C and ageing time of 2 Hours, the Hv value was 546.67 Hv and at pre-ageing temperature of 300^{o}C and ageing time of 2 Hours the Hv was 779 Hv, corresponding to increase of 42.50% in Hv.

ii. the observed microhardness at pre-ageing temperature of 300^{o}C and soaking time of 2 Hours was 779, while at ageing time of 8 Hours the value was 831.67Hv; this corresponds to an increase in Hv of 6.76%.

iii. vicker's hardness increases with increase in pre-aging temperature and aging time.

iv. DOE based on 2 level factorial design indicated that all the factors A, B, C, and D all had significant contributions; moreover, factors B and C with Prob > F of 0.0006 and 0.0009 respectively on the ANOVA Table were statistically more significant.

v. the interaction of BC, BD and CD were found to be significant.

vi. optimization of the process parameters suggest that optimum microhardness could be achieved at lower ageing time of 7 hours and reduced temperature of 470^{o}C.

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